

**Final Report**  
**Materials, Structures, and Devices**  
**for High-speed Electronics**

**NASA Grant NAG-3-154**

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# **Final Technical Report**

## **NASA Grant NAG-3-154**

Because of the length of time covered by this grant (over 10 years), a great deal of research on a number of related topics was performed. This report summarizes each area of research, by topic and time period.

1981-1984:

### **Si<sub>3</sub>N<sub>4</sub> on GaAs, Null Ellipsometry**

The initial period of the grant was devoted to using multiple wavelength and angle of incidence null ellipsometry, to characterize Si<sub>3</sub>N<sub>4</sub> thin films on GaAs substrates [1-8]. The setup included an ion laser with selectable discrete lines in the near-uv and visible, and a variable angle base. It was shown [9] that measurements at multiple wavelengths, even if only two or three, were vastly superior to single wavelength ellipsometry, in reducing correlations between model parameters (*e.g.* between film thickness and refractive index). This technique was used to characterize the structure and composition of Si<sub>3</sub>N<sub>4</sub> films as a function of depth. An effective medium approximation (EMA) was used to model the dielectric function ( $\epsilon$ ) of the film, as a mixture of pure Si<sub>3</sub>N<sub>4</sub>, amorphous silicon, voids, and SiO<sub>2</sub>. In addition, the model for the film was "sliced" into several layers, to account for differences in composition at the interface, surface, and internal parts of the film. Results for sputtered films included: 1) no measureable sign of a-Si anywhere in the film, and 2) a small fraction of SiO<sub>2</sub> throughout the film, with a higher fraction near the surface. Strong correlation between SiO<sub>2</sub> and void content prevented an unambiguous determination of the latter, but Auger electron spectroscopy confirmed the presence of oxygen and higher oxygen content near the surface. The interface between film and substrate is extremely important for device applications, and so was studied closely. Enhanced interface sensitivity was obtained by choosing an appropriate film thickness.

1984-1989

### **Diamondlike Carbon Films**

A second dielectric material studied extensively for its potential applications in microelectronics was "diamondlike carbon" (DLC), a hydrogenated form of amorphous

carbon. The optical absorption, dielectric properties, and hardness were investigated as functions of deposition parameters [10,11]. Interfacial electronic energy state density at the interface of InP was studied, to determine the suitability of DLC as the insulator in metal-insulator-semiconductor structures [12,13]. Another useful property studied was the resistance of DLC to moisture penetration, with useful applications in protective hermetic coatings [14,15]. Further complementary research on DLC properties, including optical constants, and resistance to acids and other chemicals, was conducted under NASA Lewis Grant NAG-3-95.

1983-1985:

### **VASE Development**

Some controversy had existed since the mid-1970's, over the relative merits of multiple wavelength/multiple angle of incidence null ellipsometry, and automated spectroscopic ellipsometry. The latter acquired much more data, and was much faster, but was practiced at that time at a single, fixed, nonoptimized angle of incidence. The controversy was effectively ended by development at UNL of a variable angle spectroscopic ellipsometer (VASE), which combined multiple, variable angle settings with spectroscopic measurements. The angle of incidence could be chosen for optimum sensitivity to a particular sample's structure, and confidence limits were further improved by fitting spectroscopic data measured at more than one angle. The salary of the postdoc who developed the system was born by the NASA grant, and the final product was immediately applied to the goals of the grant. The optimum sensitivity feature in particular was important in obtaining many of the research results summarized in this report.

A small spin-off company (J.A. Woollam Co.) was later formed to produce and market VASE instruments. It successfully competed internationally with other ellipsometry manufacturers, leading to the more widespread acceptance and use of angle variability. Another major factor in the market success of this new technology was the experience in applying VASE and modeling the data, gained by UNL staff working on the NASA grant and other research projects, and transferred to the company in its early stages.

1985-1988:

### **GaAs-AlGaAs Heterostructures**

One of the first uses of VASE was for characterizing epitaxially grown heterostructures [16-21], used to make very high speed transistors. The device properties depended on the thicknesses and compositions of layers only a few tens of nanometers thick. It was desired, therefore, to determine these parameters accurately, quickly, and nondestructively.  $\text{Al}_x\text{Ga}_{1-x}\text{As}$  has optical constants dependent on  $x$ , the molar fraction of Al ( $0 \leq x \leq 1$ ). Using published data for the dielectric functions of AlGaAs at discrete  $x$  values, an algorithm was developed [22] to calculate  $\epsilon$  for arbitrary  $x$ . This was an important step in the application of VASE to heterostructure characterization, since it allowed alloy composition as well as layer thicknesses to be extracted from the data (the algorithm has since been applied to other alloys, such as SiGe, InGaAs, and HgCdTe). A large number of molecular beam epitaxially (MBE) grown structures, with individual layer thicknesses ranging from 5 to 250 nm, were characterized. Samples were obtained from Perkin Elmer Corp., Honeywell Corp., University of Illinois, Wright Patterson AFB, Cornell University, McDonnell Douglas Corp., and others. Using an optimal angle near  $75^\circ$ , layer thicknesses were determined with typically less than 1 nm uncertainty, and  $x$  to within typically 0.01. VASE thickness results were compared directly with destructive XTEM and RBS measurements, with excellent agreement [18,19]. Since these were still the early days of MBE, the VASE results were often significantly different from the "nominal" values given by the crystal grower!

Another important application of VASE was to determine the lateral uniformity of layer thicknesses and compositions across the wafer [20]. Obtaining lateral uniformity was a significant problem in early MBE growth. Variations of less than 1% could be detected by VASE.

Though the primary results from VASE were layer thicknesses and composition, more subtle effects were also studied. For example, the data were sensitive to very thin native oxide and roughness overlayers [23]. Another effect was discovered, due to the enhanced sensitivity with the optimal incidence angle. A weak Franz-Keldysh effect, due to the built-in electric field (or band bending) in the heterostructure, was observed at the bandgap energy of the AlGaAs [17]. The effect had not been observed before in spectroscopic ellipsometry studies of these materials. It was subsequently studied in more detail, leading to semiquantitative analysis of the built-in fields, and doping concentrations [24-26].

1985-1987:

### **Ta-Cu Diffusion Barrier Films on GaAs**

Gold electrical contacts to GaAs are subject to interdiffusion at moderately high temperatures. Polycrystalline metallic diffusion barriers are ineffectual, because of Au diffusion along grain boundaries. Therefore amorphous  $\text{Ta}_x\text{Cu}_{1-x}$  metallic layers were investigated, as diffusion barriers for GaAs/AlGaAs high speed electronic devices. Specifically, the high temperature stability against recrystallization and chemical reaction with neighboring elements was studied [27-30]. Thin layers of amorphous  $\text{Ta}_x\text{Cu}_{1-x}$  (with a series of x values up to 0.93) were sputtered onto GaAs polished substrate wafers, and gold evaporated on top. These structures were then annealed to a series of temperatures up to 960°C to investigate the conditions under which a-TaCu crystallizes and/or chemically reacts with Au and/or Ga or As. The a-TaCu films were found to be, especially for high x values, remarkably stable up to 800°C under 30 minute vacuum anneals.

1988-1989:

### **GaAs-AlGaAs Superlattices and Multiple Quantum Wells**

Superlattices differ from heterostructures, in that the electronic and optical properties of the very thin layers are not the same as bulk. The goal of this work was to measure the optical properties of the superlattice layer, with particular attention to the quantized intersubband transitions, near the fundamental gap. These excitonic transitions have energy positions determined by the superlattice parameters: well (GaAs) and barrier (AlGaAs) thicknesses, and barrier compositions (x). The dielectric functions were obtained from VASE measurements, clearly showing the excitonic features [31-33]. One advantage of VASE over other techniques is that both the real and imaginary parts of the dielectric function are obtained, without a Kramers-Kronig analysis. Many samples, with varying superlattice parameters, were analyzed. Multiple quantum well (with thicker barriers than the superlattices) were also studied, including an asymmetric triangular MQW [34].

Advances in software accompanied these studies. Periodic structures could be modeled by defining a single "cell", with upper and lower sublayers, and upper and lower interfacial layers, and then specifying the total number of cells. (Present-day Woollam Co. software permits virtually an unlimited number of sublayers to be included in the cell).

An optoelectronic device based on the MQW, a polarization modulator, was proposed and theoretically modeled. Similar in structure to intensity modulators being investigated by others at the time, it made use of the electric field dependence of the excitonic transitions (quantum confined Stark effect). Unlike the intensity modulators, this device modulated the polarization of a beam reflected from its surface. The study involved detailed modeling of the MQW optical properties as a function of electric field, well width, barrier height, total MQW thickness, and other parameters [35].

1988:

### **Superconductivity**

In this year the grant also covered research on thallium-based high  $T_c$  superconductor deposition and characterization. A laser ablation deposition chamber was set up, with rotating target and heated substrate. The Nd:YAG laser was operated frequency doubled (520 nm). TlBaCaCuO films with  $T_c$  onset as high as 125K (2223 phase) were prepared. The films routinely had transitions to zero resistance well above 100K. In the following year, the superconductivity work was split off as a separate grant program.

1988-1991:

### ***In situ* Elevated Temperature Measurements of III-V's**

As part of the overall objective of using *in situ* SE to monitor heteroepitaxial III-V growth, the optical constants of GaAs and other III-V's as a function of temperature were needed. A UHV chamber was fitted with a spectroscopic ellipsometer, and measurements were made at temperatures from 25°C to 600°C [36-39]. An initial difficulty was related to the native oxide of GaAs. This is actually a complex mixture of several different oxides, each of which evaporates at a different temperature. It was found that the oxide desorbed at about 580°C, as expected from literature reports, but that the surface was seriously degraded in the process [36,37]. The problem was eliminated by obtaining MBE grown, arsenic capped GaAs samples. Arsenic capping protected the sample from oxidation during transportation to our chamber, where it was evaporated at about 350°C, leaving a clean unoxidized sample [38]. The upper temperature range was still limited to about 600°C, because of the excessive loss of arsenic from the surface at higher temperatures (our chamber did not provide for arsenic overpressure). With this technique,

the optical properties of GaAs as a function of temperature were measured and published. Later, the same technique was applied to AlAs [39].

An additional benefit of this work was the development of an algorithm to determine surface temperature from SE measurements [38]. The critical point energies ( $E_1$ ,  $E_1+\Delta_1$ ,  $E_2$ ) are temperature dependent, shifting to lower energies with increasing temperature. The algorithm simply interpolated between the  $\epsilon$  data measured at discrete temperatures, to get a best fit to data measured at an unknown temperature. This technique worked well compared with conventional optical pyrometry. Furthermore, its sensitivity *increased* at lower temperatures, where pyrometry becomes *less* sensitive. Thus it has potential use in such areas as low temperature III-V growth, where the growth temperature is only about 200°C.

1989-1991:

#### **Optical Constants of Thermodynamically Stable InGaAs**

Increasingly, designers of high electron mobility transistors are turning from GaAs/AlGaAs, to InGaAs on InP and GaAs, because InGaAs (the active conduction channel) has a higher electron mobility than GaAs. It is desirable to apply VASE to characterize these multilayer heterostructures, in the same way we did for GaAs/AlGaAs structures. This requires a data base of dielectric functions for  $\text{In}_x\text{Ga}_{1-x}\text{As}$  as a function of composition  $x$ . InGaAs layers of several compositions were epitaxially grown on InP and GaAs substrates, by MBE and MOCVD. The layers, which were mismatched to the substrates, were made thick enough so that the strain was relieved by misfit dislocations, resulting in thermodynamically stable material (a resulting difficulty was with surface quality of the InGaAs layers, which were roughened in a cross-hatch pattern by the misfit dislocations). The dielectric functions were determined from VASE measurements, and the algorithm used for interpolating between  $\epsilon$  of discrete AlGaAs compositions was modified for the InGaAs system [40]. These dielectric function data were then used to characterize heterostructures containing thin InGaAs layers [41,42].

1990-1992:

#### **Doping Dependence of Optical Constants of GaAs**

Commercially prepared n-type GaAs wafers were obtained, with a wide range of doping concentrations. The dielectric functions were measured, accounting for the oxide overlayer [43]. Most of the dependence on concentration was in the  $E_1$ - $E_1+\Delta_1$  doublet



region, which showed significant broadening and a slight energy shift with increasing doping. With these data, it is now possible to characterize thin GaAs layers for doping, as well as thickness.

At about the same time as this work was concluding, a new Woollam Co. VASE instrument was acquired. It extended the spectral range beyond the previous limit (840 nm, just above the GaAs bandgap energy) to one micron, giving us access to the GaAs bandgap region for the first time. Doping effects are even stronger in this region, and they also are indirectly responsible for electric field (Franz-Keldysh) effects. Work has continued on studying the bandgap region optical properties as a function of doping.

1989-1992:

### ***In-situ* Ellipsometric Studies of III-V Epitaxial Growth**

The next logical step, after amassing data and experience from *ex-situ* heterostructure and MQW characterization by spectroscopic ellipsometry, was to apply the technique *in-situ*, during growth of the structures. This work was performed by the spin-off company, which developed the *in-situ* hardware, and collaborated with an MBE researcher (G.N. Maracas, Arizona State University) [44-46]. The instrument could collect data at up to a half dozen wavelengths in quasi-real time, and could also take a full spectral scan over a longer period. The real time data clearly exhibited all of the growth processes: ramping from room to growth temperature, oxide desorption, AlGaAs and GaAs epitaxial growth. They were also analyzed for temperature, layer thicknesses, and AlGaAs composition. These initial studies demonstrated the usefulness of *in-situ* ellipsometry to epitaxial growth, while also pointing to the need for further development of hardware and analysis software.

1990-1992:

### **Photo-Thermal Spectroscopy**

An apparatus to make PTS measurements was constructed. Applications of the technique include subsurface defect imaging, thermal diffusivity measurements, and optical absorption coefficient measurements. Of these, we found the most useful to be thermal diffusivity measurements of bulk and thin films [47-49]. A new and elegant theory of the heat propagation in thin films using Green's functions was presented in a seminal publication [49]. This theory was combined with the Marquardt-Levenberg algorithm for

fitting experimental data. Recently, the strong dependence of the thermal conductivity on film thickness was the topic of a Ph.D. dissertation by William A. McGahan.

1989-1990:

#### **Micro-ellipsometry**

In many applications, one would like to use a very small beam size for ellipsometric measurements. A short project to demonstrate the feasibility of focussing the ellipsometer beam was conducted. A multiple wavelength helium neon laser beam was spatially filtered, expanded, and focused down to a spot size of between 50 and 100 microns. Measurements on a standard large-area sample were compared with conventional large-area ellipsometry measurements, and excellent agreement was found. The instrument was then used to measure small laser-annealed spots on heavily doped GaAs [50]. Though the demonstration was successful, the small number of wavelengths available from the laser source precluded spectroscopic measurements, thus greatly limiting applications. Focusing (to about 100 microns) has recently been developed at the J.A. Woollam Co. as an option for its standard 250-1000 nm VASE instrument.

1992-1993:

#### **Si Passivation, and Si/SiGe Strained-Layer Superlattices**

Aqueous HF etching of Si surfaces removes the surface oxide and terminates the Si surface with atomic hydrogen, passivating it. *Ex-situ* and *in-situ* VASE was used to monitor the passivated surface [51]. *Ex-situ* measurements determined the length of time that the passivation remained chemically stable, and resistant to oxidation. No measurable changes in the surface were observed for over 2 hours, and very little reoxidation took place over the next 3 days. After 2 months, the surface was fully reoxidized. *In-situ* measurements of the passivated surface in a UHV chamber indicated that the H-termination was desorbed after heating to 550°C. There was also evidence of slight roughening (1-2 monolayers) of the Si surface following desorption. This was the first use of *ex-situ* and *in-situ* real-time ellipsometric measurement of the surface passivation overlayer, its reoxidation rate, and surface roughness of H-terminated Si surfaces.

The Si/Si<sub>1-x</sub>Ge<sub>x</sub> strained-layer superlattice plays an important role in Si-based fast electronic devices. UHV-CVD grown superlattices, with 10 periods containing nominally 20 nm each of Si and SiGe and a Si top layer, were HF passivated and measured by SE [52]. The optical constants of SiGe were modeled from data at discrete x values, using

the same algorithm developed for AlGaAs (see above). The superlattice modeling feature of the software (see above) was employed. Allowing the individual Si and SiGe sublattice layers, the SiGe composition, the top Si layer, and the H-terminated surface layer thickness all vary, excellent fits to the SE data were obtained. Thicknesses and compositions were in excellent agreement with high-resolution x-ray diffraction and XTEM results.

## CONCLUSION

Tremendous advances in materials, devices, and instrumentation have taken place over the course of this grant. It began with *ex-situ* null ellipsometric measurements of simple dielectric films on bulk substrates. Today we are using highly automated and rapid spectroscopic ellipsometers for *ex-situ* characterization of very complex multilayer epitaxial structures. Even more impressive is the *in-situ* capability, not only for characterization but also for the actual control of the growth and etching of epitaxial layers. Spectroscopic ellipsometry has expanded from the research lab, to become an integral part of the production of materials and structures for state of the art high speed devices. Along the way, it has contributed much to our understanding of the growth characteristics and materials properties.

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## Final Equipment Report

### NASA Grant NAG-3-154

Multifrequency LCR meter, HP 427	9,260
Ultraviolet ultracollimator LHA	633
HP computer 9836A w/option 71	12,982
HP impedance analyzer	11,590
Serial impact printer	83
HP printer 82905B	795
Power supply 602B-15N	395
Winchester disk drive 20NM, HP 9134H	1,793
Counter reference	553
Photon counting photomultiplier C 1050-01	1,482
Stepper motor drive GMA 301-4	1,881
Plotter 2-pen HP 7440A	1,001
Power supply 602B-15N	401
Shutter M845	750
DAS-16 interface	955
Shutter 846	308
Shutter 846	308
Nitrogen regulator	250
Optical breadboard 77-140-02	1,845
Lock-in amplifier 3981	2,495
Light beam chopper 230	1,195
Power supply 68806	1,461
Power supply 68806	1,461
Terbium target	790
Cooled PMT housing, TE104RE	2,106
IBM XT compatible, monitor, printer	1,448
DAS-16 interface and library	1,555
Neutral density filters	762
IEEE 488 interface	565

Total: 61,103

Note: these charges do not include shipping and handling costs, which were also charged to the account.

## **Report Pursuant to Patent Rights**

### **NASA Grant NAG-3-154**

A patent disclosure was submitted for a concept of a new optoelectronic device, to the University of Nebraska Patent Administrator. The first page of the form, describing the device concept, is attached. (Some discussion of the device is also included in the enclosed Final Report for the Grant). Page 9 of the form acknowledges NASA Lewis Grant NAG-3-154 for partial support of the work on the concept. The concept was not developed into an actual device or prototype, due to the absence of the necessary growth facilities at UNL, nor was it developed elsewhere. There are no plans at present to obtain a patent for the concept.

Paul G. Snyder

3/20/89

FORM 1, ATTACHMENT 1

UNIVERSITY OF NEBRASKA

DISCLOSURE FORM

TITLE; Multiple Quantum Well Polarization Modulator

PERSONS CONTRIBUTING TO  
DEVELOPMENT OF CONCEPT:

DATE: 3/20/89

SHEET 1 of 11

Paul G. Snyder, Craig Herzinger, John A. Woollam

- I. Description and/or Sketch ( what is the concept, what does it do, how and why it works, range of applicability, etc.).

The optoelectronic device modulates the polarization of an incident light beam, when a voltage is applied across the device. The material used for the device (see diagram on page 2) is made of thin alternating layers of two different semiconductors, forming a multiple set of Quantum Wells. The electro-optic effect responsible for the polarization modulation is called the Quantum Confined Stark Effect. A schematic of the detection system, which converts the polarization changes into intensity changes, detected by optical detectors D1 and D2, is shown on page 3. The variable phase-delay compensator (C) is used to bias the output polarization to circular polarization, when no voltage is applied. Thus with no voltage, the differential output of the amplifier is zero. Applying a voltage to the PM changes the reflected polarization leading to a nonzero amplifier output.